INTERMETALLIC GROWTH AND INTERDIFFUSION IN THE Mg-Nd SYSTEM

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Abstract

Magnesium alloys with rare earth additions have exhibited high strength at room and elevated temperatures and good creep resistance largely due to their age hardenability. This study utilizes solid diffusion couples of pure magnesium and neodymium to observe the formation and growth of intermetallic phases and the interdiffusion behavior of the system. Diffusion anneals were carried out between 400 and 500°C. The resulting microstructures were examined with a scanning electron microscope and concentration profiles across the interdiffusion zone were determined via electron microprobe analysis. Two intermetallic phases were identified, $Mg_{41}Nd_5$ and Mg_3Nd_5. The MgNd phase was not observed in any of the diffusion couples. Parabolic growth constants and activation energies for growth were calculated for each phase. The $Mg_{41}Nd_5$ phase had higher growth constants. Activation energies for the growth of the intermetallic phases were calculated to be 79 kJ/mol for the $Mg_{41}Nd_5$ phase and 120 kJ/mol for the Mg_3Nd phase.

Introduction

Mg is the lightest weight structural metal available, thus making use of its alloys is highly desirable. Rare earth (RE) additions (Nd, Gd, Pr, Dy, etc.) have been shown to improve the elevated temperature properties in Mg alloys [1, 2]. Elevated temperature properties are important for applications where the stability of a component at high temperature is needed for performance, for example in automotive or aerospace applications. Diffusion in materials plays a vital role in material behavior and response to external conditions for example, in solidification, precipitation, corrosion, creep, oxidation, etc. Reliable fundamental diffusion parameters in Mg based systems are scarce. Phase development and interdiffusion in the Mg-Dy, Mg-Nd and Mg-Pr systems were studied via diffusion couples at 500°C by Xu, Chumbley, Weigelt, and Laabs [3]. In this study, phase formation and growth in the Mg-Nd binary system was observed utilizing solid-to-solid diffusion couples in the temperature range of 300-400°C.

Analytical Framework and Experimental Procedure

For diffusion-controlled growth of a phase with a semi-infinite boundary condition, thickness of the growing phase after time t, of annealing can be described by [4]:

$$k_p = \frac{x^2}{2t} \tag{1}$$

where x is the thickness of the layer and k_p is the parabolic growth constant. Typically, the temperature dependence of the parabolic growth rate constant follows the Arrhenius relation:

$$k_p = k_o \exp\left[\frac{-Q_k}{RT}\right] \tag{2}$$

where R (J/mol-K) is the ideal gas constant, Q_k is the activation energy (J/mol), and T is the

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annealing temperature in Kelvin.

Rods of pure Mg (99.9%) 7.9 mm in diameter and pure Nd (99.9%) 12.5 mm in diameter, both from Alfa Aesar™, were sectioned into 3 mm thick disks. The disk specimens were polished using a non-oxidizing lubricant down to 1 μm. Water was avoided for the entire preparation procedure for both sets of disks due to the high reactivity of both metals. The disks were then assembled in stainless steel jigs, between two inert alumina spacers with the polished faces in intimate contact as illustrated in Figure 1. The entire diffusion couple assembly is then placed in a quartz capsule which is evacuated to approximately 10^{-4} Pa (10^{-6} torr) and flushed with hydrogen and ultra high purity argon at least three times. The capsule is then backfilled with a mixture of ultra-high purity argon and hydrogen (<10%) to a pressure slightly above 10^5 Pa (1 atm) at the diffusion anneal temperature.

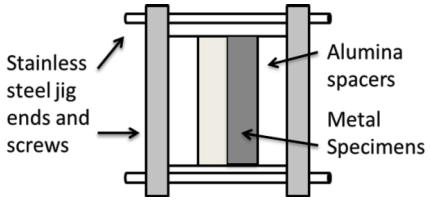

Figure 1: Schematic illustration of the diffusion couple jig assembly.

The encapsulated assembly is placed in a Paragon Bluebird™ furnace, pre-heated to the annealing temperature with an independent type-K thermocouple located near the capsule used to stabilize the furnace temperature to within ±2°C. After annealing, the capsule is quickly quenched in cold water. The entire diffusion couple jig is mounted in epoxy and then cross-sectioned with a diamond wafering blade. The cross-sections are then polished and examined with an optical microscope (OM) to check the bond quality and a scanning electron microscope (SEM) equipped with X-ray energy dispersive spectroscopy (XEDS) (Zeiss™ Ultra 55) to determine the phases present in comparison to the phase diagram shown in Figure 2. Concentration profiles are determined using electron microprobe analysis (EPMA) (JEOL™ Superprobe 733) utilizing a point-to-point scan with a 5 μm step size and a beam voltage of 20 kV and pure elemental standards.

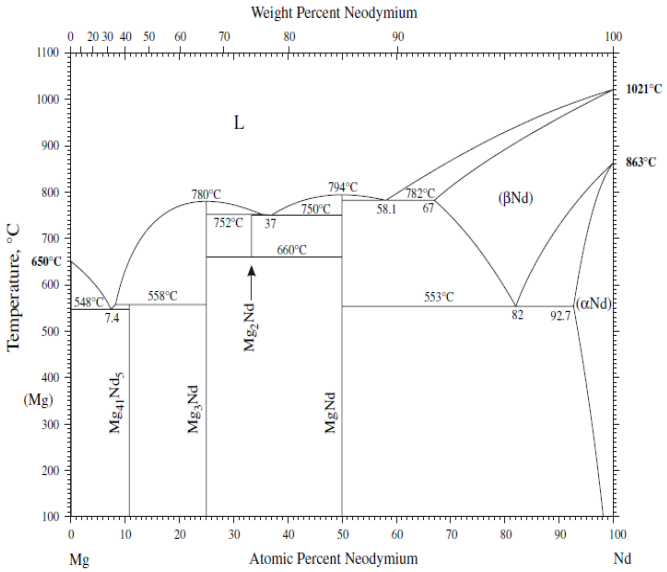

Figure 2: Equilibrium phase diagram of the Mg-Nd system [5].

Results and Discussion

The resulting diffusion microstructures are shown in Figure 3. Two distinct intermetallic layers are visible and were identified using the phase diagram, XEDS and EPMA as $Mg_{41}Nd_5$ and Mg_3Nd. Figure 4 presents the corresponding concentration profiles obtained via EPMA. From the profiles, it is evident that the $Mg_{41}Nd_5$ phase has very limited, if any, solubility, while the Mg_3Nd phase exhibits significant solubility, especially at higher temperature.

Using the micrographs in Figure 3, thickness measurements of each intermetallic phase were made using image analysis at 15 random locations per phase, per diffusion couple. These thickness measurements were then used in conjunction with Eq. 1 to calculate parabolic growth constants, as reported in Table 1. The pre-exponential factor and activation energy for growth of each phase were determined using Eq. 2 and are also reported in Table 1. The temperature dependence of the parabolic growth constants for both phases is shown in Figure 5. It is evident from both Figure 5 and Table 1 that the $Mg_{41}Nd_5$ phase has higher growth constants and lower activation energy, which agrees with the larger thickness observed for this phase as seen in Figure 3.

The $Mg_{41}Nd_5$ phase grew the thickest and correspondingly displayed the fastest growth constants and lower activation energy for growth despite being the lower temperature phase. The highest temperature phase MgNd, however, was not observable in any of the diffusion couples. This could possibly be due to unfavorable nucleation conditions for this phase, or the surrounding phases having faster diffusion coefficients than the MgNd phase.

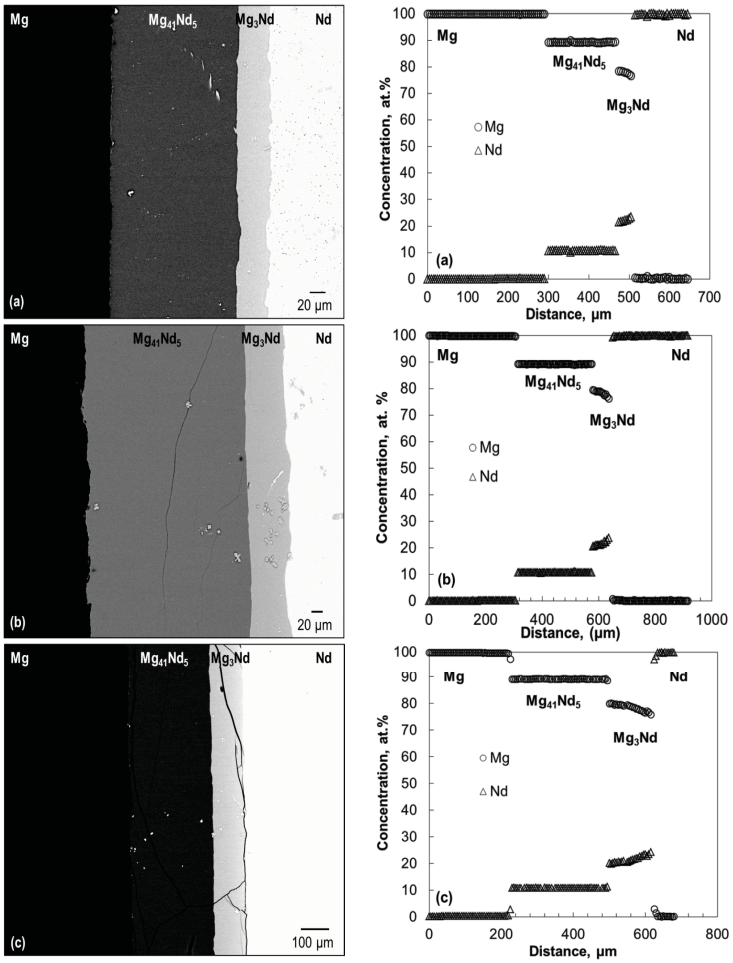

Figure 3: Backscatter electron micrographs of the Mg vs. Nd diffusion couple cross- sections at (a) 400°C for 21 days, (b) 450°C for 15 days, and (c) 500°C for 10 days.

Figure 4: Concentration profiles determined using EPMA for each diffusion couple.

Table 1: Phase layer thickness measurements, parabolic growth constants, pre-exponential factors and activation energies for growth for the Mg_3Nd and $Mg_{41}Nd_5$ intermetallic phases.

Diffusion couple	400°C 21 days		450°C 15 days		500°C 10 days	
	$Mg_{41}Nd_5$	Mg_3Nd	$Mg_{41}Nd_5$	Mg_3Nd	$Mg_{41}Nd_5$	Mg_3Nd
Y (μm)	173.6	43.6	267.4	65.5	296.2	121.3
k_p (m^2/s)	$8.3x10^{-15}$	$5.2x10^{-16}$	$2.7x10^{-14}$	$1.7x10^{-15}$	$5.1x10^{-14}$	$8.5x10^{-15}$
	$Mg_{41}Nd_5$		Mg_3Nd			
k_o (m^2/s)	$1.2x10^{-8}$		$9.6x10^{-7}$			
Q_k (kJ/mol)	79.0		120.0			

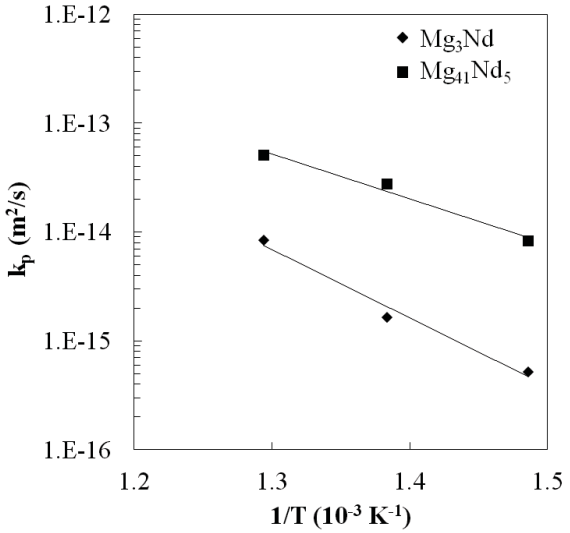

Figure 5: Temperature dependence of the parabolic growth constant for both intermetallic phases, Mg_3Nd and $Mg_{41}Nd_5$.

Summary

Three Mg vs. Nd diffusion couples were assembled and annealed at 400, 450, and 500°C for 21, 15 and 10 days respectively. Phase formation and growth were studied via optical and scanning electron microscopy and electron probe micro-analysis. The $Mg_{41}Nd_5$ and Mg_3Nd intermetallic phases were observed in all three couples, however the MgNd phase was not observed. The $Mg_{41}Nd_5$ phase was determined to have the highest parabolic growth constants and the lowest activation energy for growth and was seen to grow the thickest layer in each diffusion couple. Integrated interdiffusion coefficients were determined as well as pre-exponential factors and activation energies for interdiffusion for each phase present and will be reported elsewhere.

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References

[1] B. M. J.F Nie, "Characterization of strengthening precipitate phases in a Mg-Y-Nd alloy," Acta Mater., vol. 48, pp. 1691-1703, 2000.

[2] M. P. A. Luo, "Review Cast magnesium alloys for elevated temperature applications," J. Mater. Sci., vol. 29, pp. 5259-5271, 1994.

[3] Y. Xu, L. Chumbley, G. Weigelt and F. Laabs, "Analysis of interdiffusion of Dy, Nd, and Pr in Mg," J. Mater. Res. , vol. 16, no. 11, pp. 3287-92, 2001.

[4] J. Philibert, Atom Movements Diffusion and Mass Transport in Solids, France: Les Editions de Phusique, 1991.

[5] H. Okamoto, "Mg-Nd," J. Phase Equilibria and Diff., vol. 28, no. 4, p. 405, 2007.